The determination of Inorganic Analytes by Inductively-Coupled Plasma Mass Spectrometry

1. Introduction

The analytical method described here quantifies inorganic analytes in samples by inductively-coupled plasma mass spectrometry (ICP-MS).
Samples are nebulized and the resulting aerosol is transported by argon gas into the plasma torch. The ions produced by high temperatures are entrained in the plasma and extracted through a differentially pumped interface and separated on the basis of their mass-to-charge ratio by a mass spectrometer. The ions transmitted through the mass spectrometer are measured by an electron multiplier and the signals are processed by the instrument's data handling system. Interferences must be assessed and valid corrections applied to the raw data. Interference corrections include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

2. Interfaces with Other Methods

This method interfaces with the following EGL Methods:

- EGL Method 29, Calibration of Laboratory Scales and Analytical Balances
- EGL Method 25, Method for Sample Login, Control, and Disposition
- EGL Method 22, Trace Element Digestion

Operation of the ICP-MS instrumentation is discussed in EGL Work Instruction 04, Operation of the ICP-MS.

3. Materials and Equipment

- Deionized water (DI H_2O).
- Nitric acid (HNO_3), Trace Metal grade or better.
- Hydrochloric acid (HCl), Trace Metal grade or better.
- ICP-MS system equipped with an auto-sampler.
- Polypropylene, polystyrene, or glass test tubes (appropriate for type of analysis)
- Calibration Standards made from commercially available stock solutions

4. Procedure

Prepare stock solutions according to the appropriate work instruction. The stock solutions include rinses, calibration standards, and internal standards.
Follow the appropriate work instructions for startup of the ICP-MS and verify the instrument is running correctly and sensitivity is within manufacturer specifications.
Keep records of performance and maintenance according to EGL guidelines.

5. Calibration and Quality Control Samples

All calibration standards are prepared using the appropriate matrix matched blank solution. Calibration standards are to be prepared each day of analysis. QC standards include blanks, intermediate standard, duplicates, and standard reference materials (SRM).Calibration coefficients shall be at least 0.995 before proceeding with running samples. A digestion blank will be run at the beginning of the run along with a low intermediate check standard to verify the lower end of the calibration. For every 10 samples either a SRM or duplicate will be run. Samples with results greater than the highest calibration standard must be diluted and reran. The data will be deemed acceptable if SRM and duplicates fall within 3 sigma of the SRM value or original value of the duplicate sample.
The performance of the method is checked using digested SRM's. If the method performance falls outside EGL limits the samples are re-digested and analyzed again.

6. Limits, Precautions and Interferences

Mass interferences are well documented in many publications and most elements have multiple masses to choose from with a few exceptions.
Isobaric elemental interferences are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrometer. All data obtained under such conditions must be corrected by measuring the signal from another isotope of the interfering element and subtracting the appropriate signal ratio from the isotope of interest. This is done automatically by pre-programmed correction factors in the instrument software. The correction formulas if used are given in the instrument documentation.
Isobaric polyatomic ion interferences are caused by polyatomic species which have the same nominal mass-to-charge ratio as the isotope of interest, and which cannot be resolved by the mass spectrometer in use. These ions are commonly formed in the plasma or interface system from support gases or sample components, and are therefore highly dependent on the sample matrix and chosen instrument conditions. Such interferences must be recognized, and when they cannot be avoided by the selection of alternative analytical isotopes, appropriate corrections must be made by programming in a correction factor for the elements which are affected. The correction factors if used are given in the instrument documentation.
Physical interferences may occur in the transfer of solution to the nebulizer (e.g. viscosity effects), at the point of aerosol formation and transport to the plasma (e.g. surface tension), or during excitation and ionization processes within the plasma itself. High levels of dissolved solids in the sample may contribute deposits of material on the extraction and/or skimmer cones reducing the effective diameter of the orifices and therefore ion transmission.
Memory interferences result when isotopes of elements in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the sampler and skimmer cones as well as from the buildup of sample material in the plasma torch and spray chamber.

These interferences are mitigated by analyzing internal standards for correction of the instrument response and by analyzing blanks.

7. Acceptance of Data

For each batch of samples processed a minimum of one digestion blank, duplicate and SRM must be carried throughout the entire sample preparation and analytical process.
A digestion blank (processed per EGL Method 22) is carried through the appropriate steps of the analytical process. These steps may include, but are not limited to, pre-filtering, digestion, dilution, filtering, and analysis.
These blanks are useful in determining if digested samples have been contaminated in any part of the dissolution process.
A SRM is also carried throughout the entire sample preparation and analytical process. Acceptance criteria should be defined at a laboratory derived limit developed through the use of historical analyses. In the absence of historical data generated criteria, this limit should be set at ± 3 sigma of the recommended values.
Duplicate samples should be within ± 3 sigma of the original sample value.
If any of these criteria are not met and no reasonable explanation can be given the batch must be reprocessed and analyzed again.
The reporting limits and masses used for this method are in the appropriate work instruction.

8. Data Handling and Transfer

The sample weights and names (ID's) captured in EGL method 22 are transferred to the sample run file in the ICP-MS software and used to calculate the appropriate dilution and or back to the dry sample basis. Data is acquired and the results are put in the report folder on the instrument computer. The "name".rop file is transferred from the report folder to an Excel™[1] template electronically. A macro within the template puts the correct headers required by the EGL laboratory information management system (LIMS). The data is reviewed and saved in the data to be entered folder on the shared network drive for upload into the LIMS. All steps in data migration to the LIMS will be saved.

9. References:

None.

10. Attachments

None.

11. History of Changes

Revision 0: initial issue.

---

[1] Any use of trade names is for descriptive purposes only and does not imply endorsement by the U.S. Government.